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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁷ : C11D 3/42, 3/22, 17/06	A1	(11) International Publication Number: WO 00/58431 (43) International Publication Date: 5 October 2000 (05.10.00)
<p>(21) International Application Number: PCT/EP00/02332</p> <p>(22) International Filing Date: 16 March 2000 (16.03.00)</p> <p>(30) Priority Data: 0590/99 29 March 1999 (29.03.99) CH</p> <p>(71) Applicant (for all designated States except US): CIBA SPECIALTY CHEMICALS HOLDING INC. [CH/CH]; Klybeckstrasse 141, CH-4057 Basel (CH).</p> <p>(72) Inventors; and (75) Inventors/Applicants (for US only): REINEHR, Dieter [DE/DE]; Wolfsheule 10, D-79400 Kandern (DE). METZGER, Georges [FR/FR]; Herrenweg 228, F-68480 Moernach (FR). KRAMER, Hans [NL/CH]; Landstrasse 59, CH-5073 Gipf-Oberfrick (CH).</p> <p>(74) Common Representative: CIBA SPECIALTY CHEMICALS HOLDING INC.; Patentabteilung, Klybeckstrasse 141, CH-4057 Basel (CH).</p>		<p>(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: USE OF A BRIGHTENER PIGMENT IN CLEANSERS OR DETERGENTS</p> <p>(57) Abstract</p> <p>A description is given of the use of a brightener pigment, which comprises (a) a cellulose powder which is dispersible in water, and (b) a water-soluble fluorescent whitening agent. The brightener pigments are used for improving the aspect of detergents. The fluorescent whitening agent does not come into contact with the goods to be washed.</p>		

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Use of a brightener pigment in cleansers or detergents

The present invention relates to the use of a brightener pigment, which comprises

- (a) a cellulose powder which is dispersible in water, and
 - (b) a water-soluble fluorescent whitening agent
- for increasing the whiteness of cleansers or detergents.

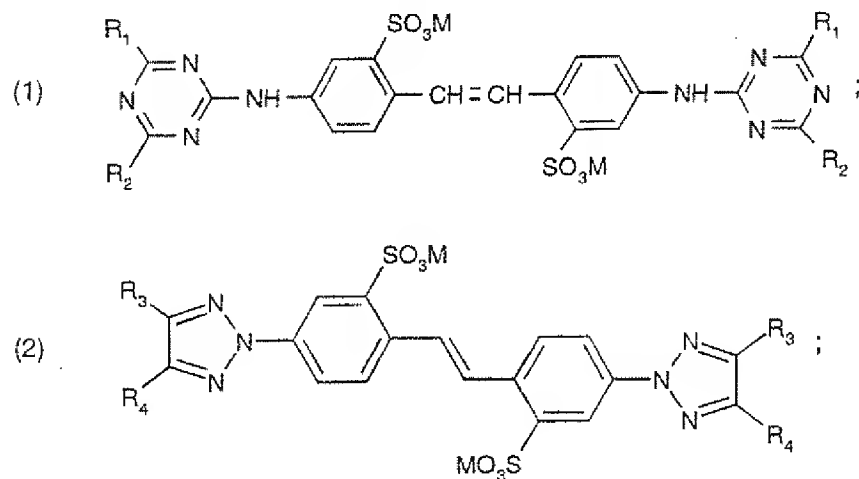
A cellulose powder suitable as component (a) is, for example, microcrystalline cellulose which is obtained from cotton linters or wood cellulose, usually by a combination of hydrolytic degradation and mechanical wet crushing of the particles with subsequent spray-drying, and which no longer has a fibre structure (viz. "Umschau" 77 (1977), 312).

Also suitable are cellulose powders which are produced by dry grinding standard wood fillers and which likewise no longer have a fibre structure.

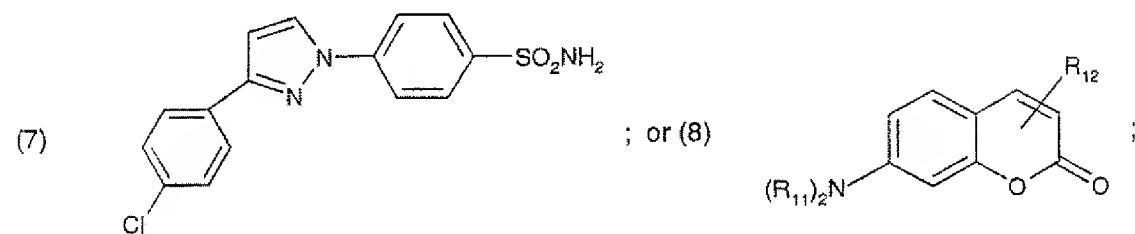
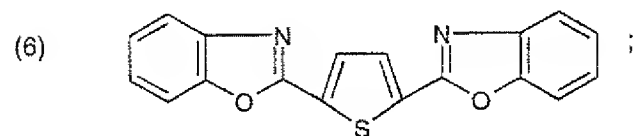
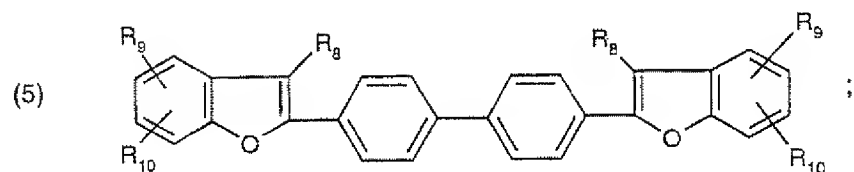
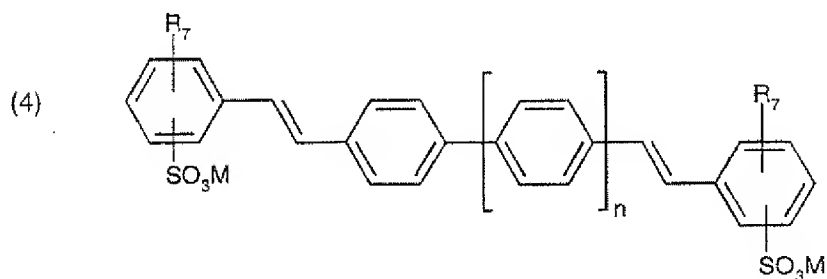
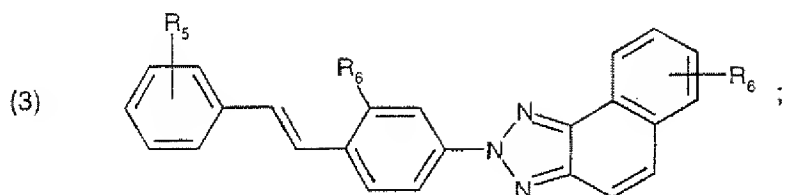
Particularly suitable cellulose powders are those having a high crystallinity of more than 30%, preferably of more than 50%, as well as an ability to reduce the hydrophilicity, and a dispersibility in water of up to a solids content of about 25.

Water-insoluble types of cellulose are also suitable, for example cellulose ethers, such as methyl- or ethylcellulose, or cellulose powders having a particle size of 25 to 35, preferably of 30 μm .

Fluorescent whitening agents suitable for use according to this invention and corresponding to component (b) conform to formula



- 2 -



wherein

R_1 and R_2 are each independently of the other -OH, -Cl, -NH₂, -O-C₁-C₄alkyl, -O-aryl, -NH-C₁-C₄alkyl, -N(C₁-C₄alkyl)₂, -N(C₁-C₄alkyl)(C₁-C₄hydroxyalkyl), -N(C₁-C₄hydroxyalkyl)₂, or -NH-aryl, morpholino, S-C₁-C₄alkyl(aryl),

R_3 and R_4 are each independently of the other hydrogen, C₁-C₄alkyl, phenyl or a radical of

- 3 -



R_5 is hydrogen; Cl or SO_3M ;

R_6 is $-\text{CN}$; $-\text{SO}_3\text{M}$; $-\text{S}(\text{C}_1\text{-C}_4\text{alkyl})_2$; or $\text{S}(\text{aryl})_2$;

R_7 is hydrogen; $-\text{SO}_3\text{M}$; $-\text{O}-\text{C}_1\text{-C}_4\text{alkyl}$; $-\text{CN}$; $-\text{Cl}$; $-\text{COO}-\text{C}_1\text{-C}_4\text{alkyl}$; or $\text{CON}(\text{C}_1\text{-C}_4\text{alkyl})_2$;

R_8 is hydrogen; $-\text{C}_1\text{-C}_4\text{alkyl}$; $-\text{Cl}$ or $-\text{SO}_3\text{M}$;

R_9 and R_{10} are each independently of the other hydrogen; $\text{C}_1\text{-C}_4\text{alkyl}$; $-\text{SO}_3\text{M}$; $-\text{Cl}$; or $-\text{O}-\text{C}_1\text{-C}_4\text{alkyl}$;

R_{11} is hydrogen; or $\text{C}_1\text{-C}_4\text{alkyl}$;

R_{12} is hydrogen; $\text{C}_1\text{-C}_4\text{alkyl}$; $-\text{CN}$; $-\text{Cl}$; $-\text{COO}-\text{C}_1\text{-C}_4\text{alkyl}$; $-\text{CON}(\text{C}_1\text{-C}_4\text{alkyl})_2$; aryl or $-\text{O-aryl}$;

M is hydrogen; Na; K; Ca; Mg; ammonium; mono-, di-, tri- or tetra- $\text{C}_1\text{-C}_4\text{alkyl}$ ammonium; mono-, di- or tri- $\text{C}_1\text{-C}_4\text{hydroxyalkyl}$ ammonium; or ammonium which is di- or trisubstituted by a mixture consisting of $\text{C}_1\text{-C}_4\text{alkyl}$ and $\text{C}_1\text{-C}_4\text{hydroxyalkyl}$ groups; and

n is 0; or 1.

$\text{C}_1\text{-C}_4\text{Alkyl}$ is a branched or unbranched radical and is, for example, methyl, ethyl, propyl, isopropyl, n-butyl and may be unsubstituted or substituted by halogen, such as fluoro, chloro or bromo, $\text{C}_1\text{-C}_4\text{alkoxy}$, such as methoxy or ethoxy, phenyl or carboxyl, $\text{C}_1\text{-C}_4\text{alkoxycarbonyl}$, such as acetyl, mono- or di- $\text{C}_1\text{-C}_4\text{alkylamino}$ or $-\text{SO}_3\text{M}$.

The compounds of formula (1) are preferably used in neutral form, i.e.

M is preferably a cation of an alkali metal, in particular sodium.

The compounds of formula (1) may be prepared under known reaction conditions by reacting cyanuric chloride with the corresponding aminostilbenesulfonic acids and with an amino compound which is capable of introducing an R_1 group and with a compound which is capable of introducing an R_2 group, R_1 and R_2 being as defined above.

Examples of the fluorescent whitening agents which are advantageously used in this invention are listed in the following Table 1:

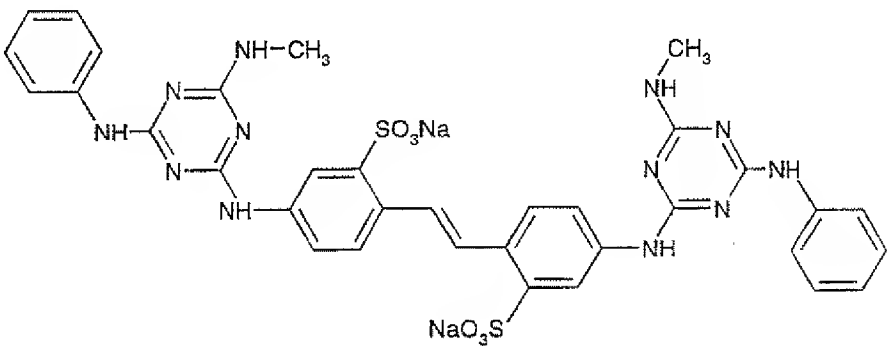
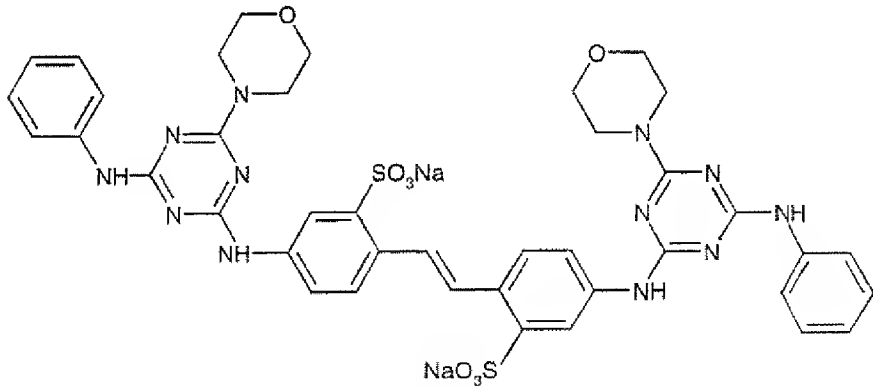
Table 1:	
compound of formula	
(9)	
(10)	

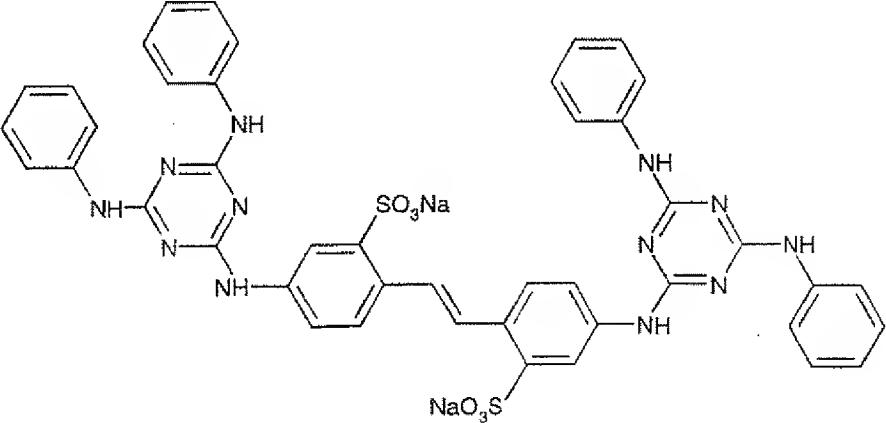
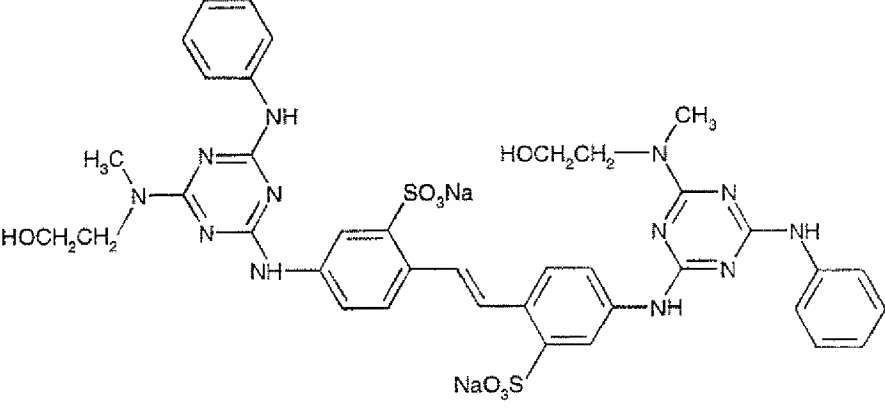
Table 1:	
compound of formula	
(11)	 <p>Chemical structure (11) is a symmetrical molecule. It features two 2,4,6-triphenyl-1,3,5-triazine-5-yl groups connected by a trans-stilbene bridge. The central double bond connects the 5-positions of the two triazine rings. Each triazine ring has phenyl groups at the 2, 4, and 6 positions. The central double bond is trans, with the SO₃Na groups on opposite sides of the bridge.</p>
(12)	 <p>Chemical structure (12) is a symmetrical molecule. It features two 2-methyl-2-(2-hydroxyethyl)amino-4,6-diphenyl-1,3,5-triazine-5-yl groups connected by a trans-stilbene bridge. The central double bond connects the 5-positions of the two triazine rings. Each triazine ring has a 2-methyl-2-(2-hydroxyethyl)amino group at the 2-position and phenyl groups at the 4 and 6 positions. The central double bond is trans, with the SO₃Na groups on opposite sides of the bridge.</p>

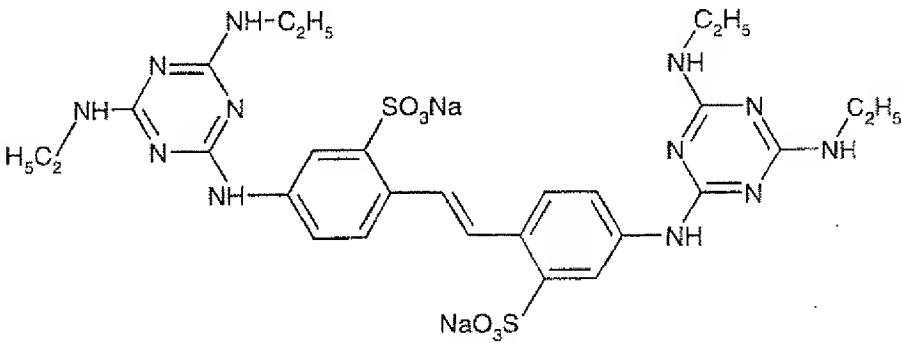
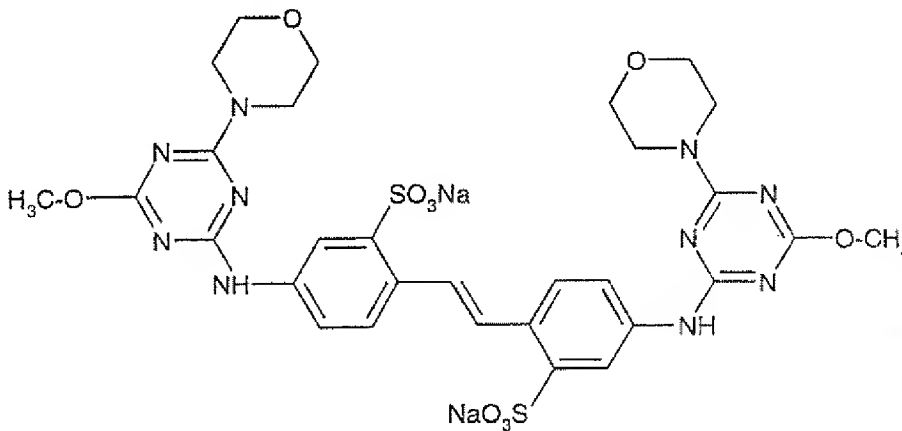
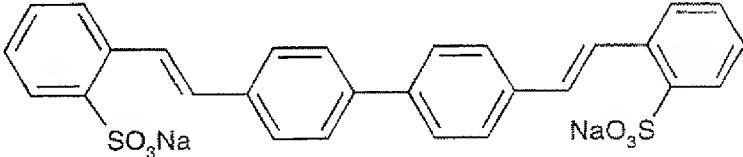
Table 1:	
<u>compound of formula</u>	
(13)	 <chem>CCNC1=NC=NC(NC2=CC=C(C=C2C=CC3=CC=C(NC4=NC=NC(NC5=CC=C(C=C5)S(=O)(=O)[O-])[Na])N4)N3)N1</chem>
(14)	 <chem>COC1=NC=NC(NC2=CC=C(C=C2C=CC3=CC=C(NC4=NC=NC(NC5=CC=C(C=C5)S(=O)(=O)[O-])[Na])N4)N1N6CCOCC6</chem>
(15)	 <chem>[Na]S(=O)(=O)C1=CC=C(C=C1C=CC2=CC=CC=C2C=CC3=CC=C(C=C3)C=CC4=CC=C(C=C4)S(=O)(=O)[Na])</chem>

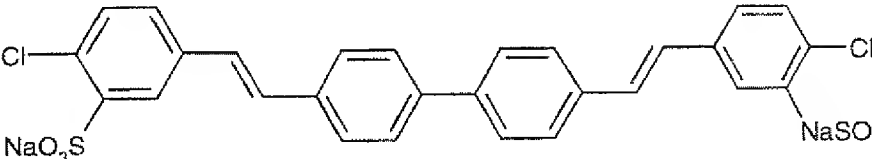
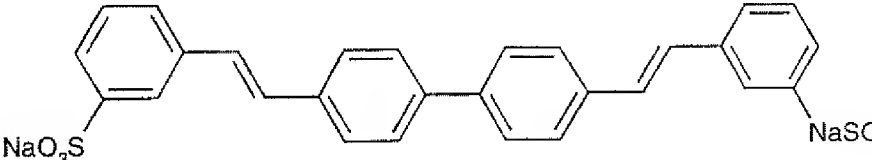
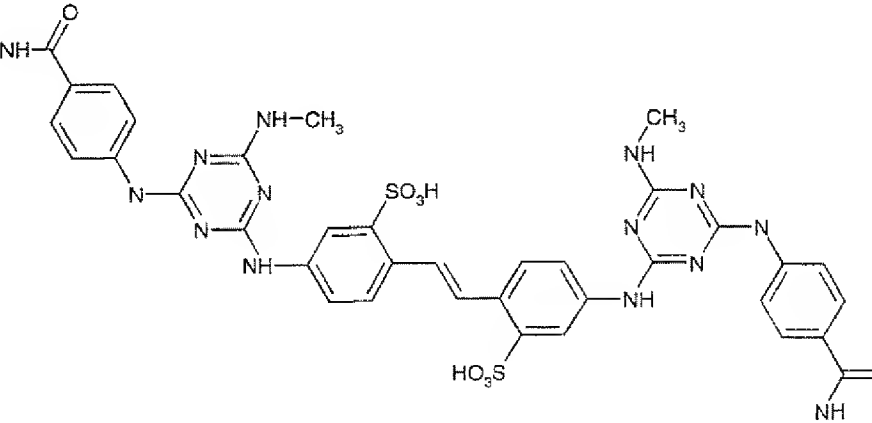
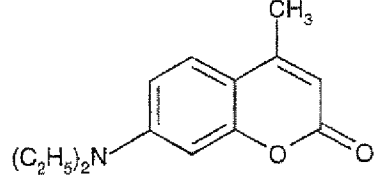
Table 1:	
compound of formula	
(16)	
(17)	
(18)	
(19)	

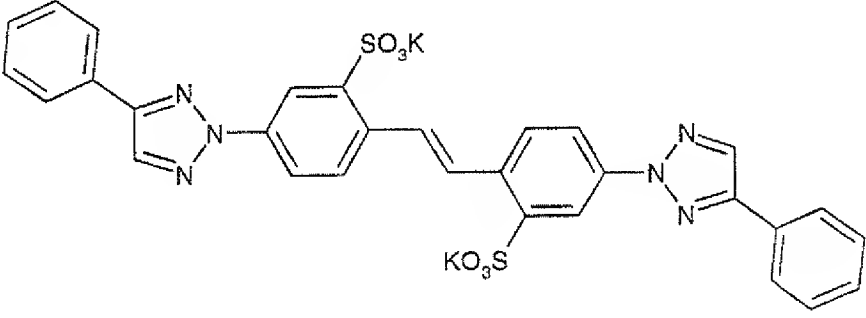
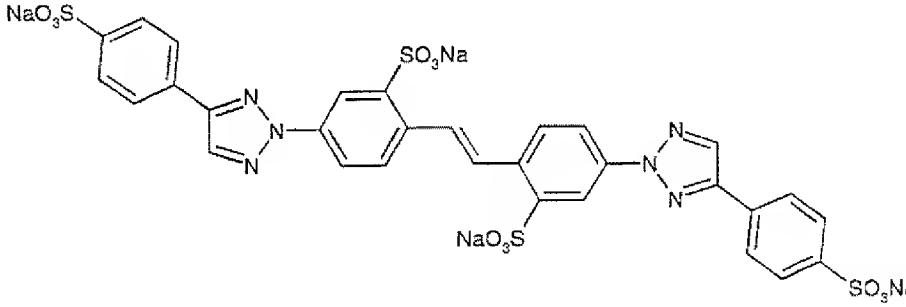
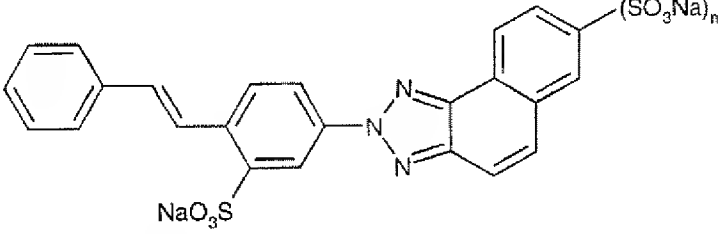
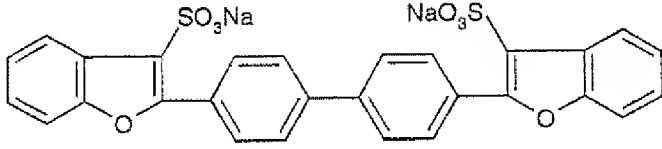
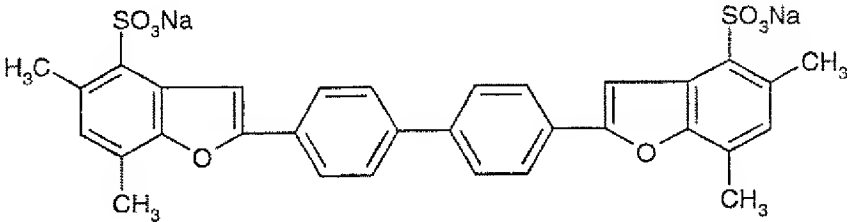
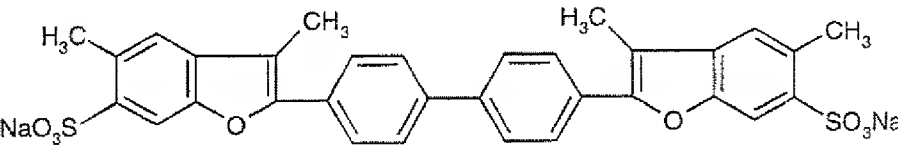
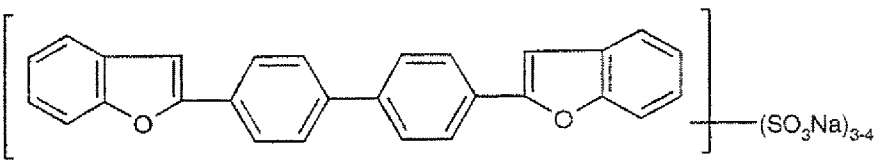
Table 1:	
compound of formula	
(20)	
(21)	
(22)	
(23)	

Table 1: compound of formula	
(24)	
(25)	
(26)	

The fluorescent whitening agents used according to this invention and corresponding to the component (b) are preferably used in amounts from 0.001 to 1.0 % by weight, more preferably from 0.01 to 0.5 and, particularly preferably, from 0.01 to 0.5 % by weight, based on the entire amount of dispersible cellulose powder used. The fluorescent whitening agents can in this case be added to the dispersible cellulose powder as individual compounds or also in the form of mixtures consisting of several individual compounds.

In principle, the brightener pigment used according to this invention and comprising the components (a) and (b) can be used singly or also together with other pigments for brightening cleansers and detergents. Other pigments used may be, for example, talcum, titanium dioxide, aluminium oxide, aluminium hydroxide, zinc oxide, chalk or clays, such as kaolin.

The brightener pigment used according to this invention may be prepared in simple manner by dispersing suitable cellulose powders in water and charging them with one or more than

one of the above water-soluble fluorescent whitening agents.

Even at a low temperature, the brightener readily adsorbs to the dispersed cellulose powder, and the brightened cellulose powder shows no greying even at relatively high brightener concentrations.

The dispersions so obtained can contain customary anionic or nonionic dispersants, for example the salts of polycarboxylic acids, sulfated or non-sulfated higher alkanols or alkyl-phenol polyglycol ethers, lignin sulfonates, carboxymethylcellulose, hydroxyethylcellulose, and the like.

The amount of fluorescent whitening agent used depends on the amount of cellulose powder used in the brightener pigments employed according to this invention. Generally, amounts of up to 10 % by weight of active substance, based on the cellulose powder, may be used. Higher amounts do not produce any additional brightening effect as the greying limit is exceeded. It is preferred to use amounts in the range from 2.5 to 7 % by weight, based on the dry cellulose powder.

Dazzling white organic brightener pigments are obtained in which the fluorescent whitening agent is incorporated or to which it is adsorbed. These products are solid colloidal particles having an average diameter of 0.1 to 0.2 μm which are agglomerated to pigment particles having an average diameter of 3 to 20 μm .

The optically brightened pigments produced in this manner are excellently suitable for improving the whiteness (aspect improvement) of commercial cleansers or detergents.

After being prepared, the brightener pigment can be added immediately in disperse form to the cleanser or detergent.

The aqueous brightener pigment can also be processed to a powder by standard process steps, such as filtration or drying.

The brightener pigment used according to this invention is preferably usually incorporated into the cleanser or detergent by first slurring the brightener pigment, with stirring, in water and then adding the corresponding cleanser or detergent with further addition of water. A creamy slurry is thus obtained which is then dried and sieved. This yields a cleanser or detergent having a particle size of about > 0.3 to 1 mm.

In another embodiment of this invention, the optically brightened cleanser or detergent may be prepared by simple powdering with the brightener pigment. To this purpose, 0.5 to 20%,

typically 1-10%, of brightener pigment, based on the component to be brightened, are mixed dry until the particles are coated with the pigment.

Suitable agents which may be treated according to this invention with the brightener pigment comprising the components (a) and (b) are cleansers or detergents in powdered or granulated form. The detergent may be composed of one or more than one granular component in the form of particles, at least one granular component being enriched with the brightener pigment.

Suitable detergents are preferably in granular form having a high bulk density. In addition to the brightener pigment, the detergent may contain further ingredients, for example surfactants, inorganic and organic builder substances, bleaching agents, substances which positively affect the oil or fat removability, greying inhibitors and, optionally, substances which enhance the solubility and rate of dissolution of the individual granular components and/or of all agents, textile softening substances, colourants and fragrances and also alkaline and/or neutral salts in the form of their sodium and/or potassium salts.

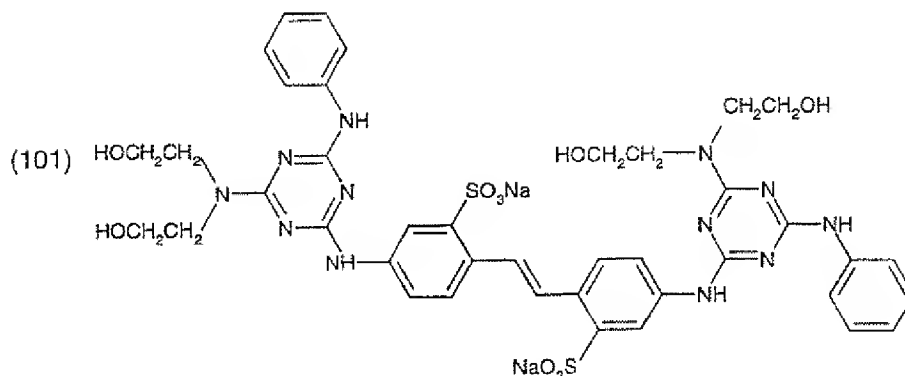
The cleanser or detergent treated in this manner is distinguished by a very high degree of whiteness which is substantially higher than when the organic white pigment and optical fluorescent whitening agent are added separately.

Another advantage of the cleanser or detergent thus enriched is that in the actual washing process the brightener pigment or fluorescent whitening agent does not contact the goods to be washed and is not absorbed by them.

The following non-limitative Examples illustrate the invention in more detail.

Example 1:

12.5 g of a cellulose powder with 0.2 % of the compound of formula



are mixed with 25 g of standard washing powder (ECE). This washing powder consists of:

- 8.0 % of sodium alkylbenzenesulfonate
- 2.9 % of tallow alcohol tetradecanethylene glycol ether 14 EO
- 3.5 % of soda soap
- 43.8 % of sodium triphosphate
- 7.5 % of sodium silicate
- 1.9 % of magnesium silicate
- 1.2 % of CMC
- 0.2 % of EDTA
- 21.2 % of sodium sulfate
- 9.8 % of water

After the brightened cellulose powder has been incorporated, the whiteness of the powder is determined by the Ganz method using a DCI/SF 500 spectrophotometer.

The addition of cellulose powder increases the degree of whiteness of the washing powder to 163 (according to Ganz). Without such an addition, the washing powder has a degree of whiteness of 51 (according to Ganz).

A washing liquor is prepared consisting of 0.4 g of the above washing agent in 50 ml of drinking water.

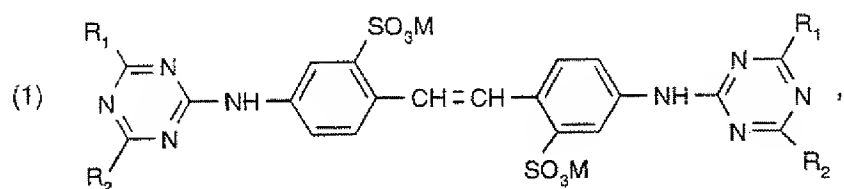
10 g of bleached cotton are placed in the liquor and washed for 15 minutes at 25°C. The fabric is then rinsed, spun and ironed at 160°C. This washing process is repeated 5 times.

After washing 5 times, the degree of whiteness of the treated fabric samples is determined.

After washing 5 times, the fabric samples washed with the washing powder/cellulose mixture have a degree of whiteness of 106, which is substantially lower than if the compound (101) is not adsorbed to the cellulose powder.

What is claimed is

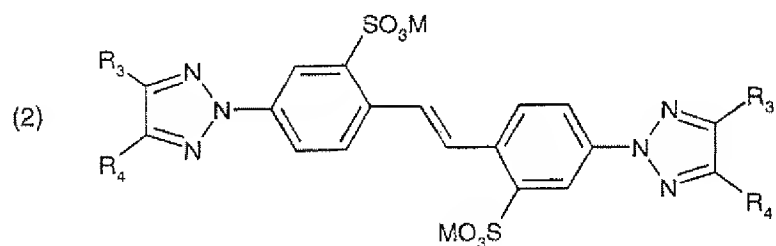
1. Use of a brightener pigment, which comprises
 - (a) a cellulose powder which is dispersible in water, and
 - (b) a water-soluble fluorescent whitening agent
 for increasing the whiteness of cleansers or detergents.
2. Use according to claim 1, wherein the cellulose powder (component (a)) has a particle size of 25 to 35 μm .
3. Use according to either claim 1 or claim 2, wherein component (b) is a fluorescent whitening agent of formula



wherein

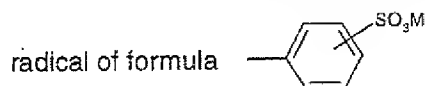
R_1 and R_2 are each independently of the other -OH, -Cl, -NH₂, -O-C₁-C₄alkyl, -O-aryl, -NH-C₁-C₄alkyl, -N(C₁-C₄alkyl)₂, -N(C₁-C₄alkyl)(C₁-C₄hydroxyalkyl), -N(C₁-C₄hydroxyalkyl)₂, or -NH-aryl, morpholino, S-C₁-C₄alkyl(aryl).

4. Use according to either claim 1 or claim 2, wherein the component (b) is a fluorescent whitening agent of formula

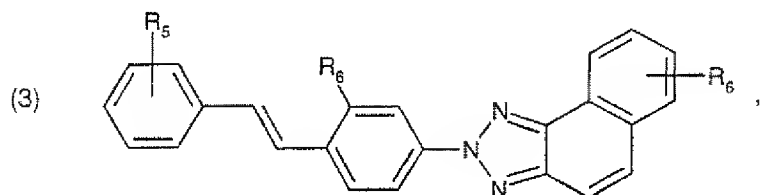


wherein

R_3 and R_4 are each independently of the other hydrogen, C₁-C₄alkyl, phenyl or a



5. Use according to either claim 1 or claim 2, wherein the component (b) is a fluorescent whitening agent of formula

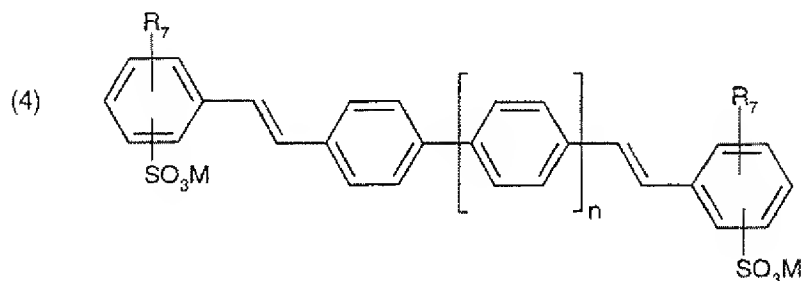


wherein

R_5 is hydrogen, Cl or SO_3M ; and

R_6 is $-\text{CN}$, $-\text{SO}_3\text{M}$, $-\text{S}(\text{C}_1\text{-C}_4\text{alkyl})_2$ or $\text{S}(\text{aryl})_2$.

6. Use according to either claim 1 or claim 2, wherein the component (b) is a fluorescent whitening agent of formula

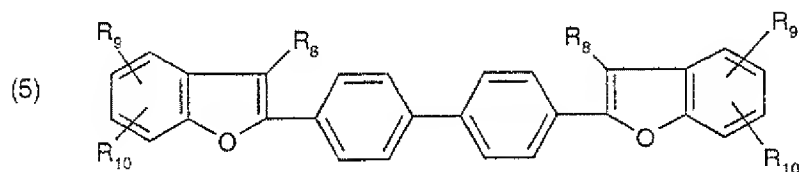


wherein

R_7 is hydrogen, $-\text{SO}_3\text{M}$, $-\text{O}-\text{C}_1\text{-C}_4\text{alkyl}$, $-\text{CN}$, $-\text{Cl}$, $-\text{COO}-\text{C}_1\text{-C}_4\text{alkyl}$, or $\text{CON}(\text{C}_1\text{-C}_4\text{alkyl})_2$, and

M has the meaning cited in claim 1.

7. Use according to either claim 1 or claim 2, wherein the component (b) is a fluorescent whitening agent of formula



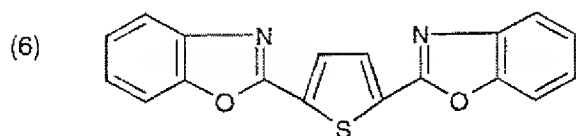
wherein

R_8 is hydrogen; $-\text{C}_1\text{-C}_4\text{alkyl}$, $-\text{Cl}$ or $-\text{SO}_3\text{M}$; and

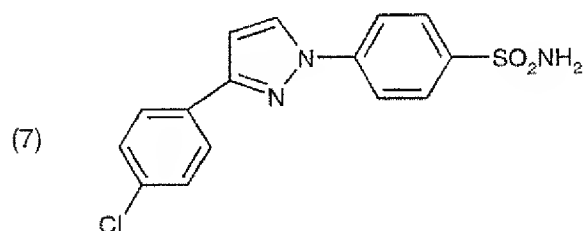
R_9 and R_{10} are each independently of the other hydrogen, $\text{C}_1\text{-C}_4\text{alkyl}$, $-\text{SO}_3\text{M}$, $-\text{Cl}$ or

-O-C₁-C₄alkyl.

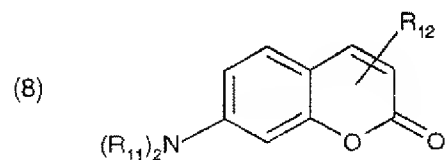
8. Use according to either claim 1 or claim 2, wherein the component (b) is a fluorescent whitening agent of formula



9. Use according to either claim 1 or claim 2, wherein the component (b) is a fluorescent whitening agent of formula



10. Use according to either claim 1 or claim 2, wherein the component (b) is a fluorescent whitening agent of formula



wherein

R₁₁ is hydrogen or C₁-C₄alkyl; and

R₁₂ is hydrogen, C₁-C₄alkyl, -CN, -Cl, -COO-C₁-C₄alkyl, -CON(C₁-C₄alkyl)₂, aryl or -O-aryl.

11. Use according to any one of claims 1 to 10, wherein the cleanser or detergent is in powdered or granulated form.

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 00/02332

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C11D3/42 C11D3/22 C11D17/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 41 40 830 A (CIBA GEIGY AG) 17 June 1992 (1992-06-17) claims page 3, line 64 - page 4, line 1 ---	1, 4-6, 11
A	EP 0 145 438 A (PROCTER & GAMBLE) 19 June 1985 (1985-06-19) claims 1-3, 6-11 page 4, line 13 - line 39 ---	1, 2, 11
A	EP 0 900 783 A (CIBA GEIGY AG) 10 March 1999 (1999-03-10) claims 1, 8-23 examples 9-21 page 3, line 8 - line 29 ---	1, 6, 11

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

20 July 2000

Date of mailing of the international search report

28/07/2000

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INTERNATIONAL SEARCH REPORT

Int. Jonal Application No

PCT/EP 00/02332

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, A	<p>W0 99 51714 A (UNILEVER)</p> <p>14 October 1999 (1999-10-14)</p> <p>claims 1-4, 12</p> <p>page 1, line 8 - line 15</p> <p>-----</p>	1, 3-11

INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/EP 00/02332

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WO 9951714	A	14-10-1999	AU 5265299 A	25-10-1999

